METHODS FOR THE ANALYSIS OF IRON AND STEEL, USED IN LABORATORIES OF THE AMERICAN ROLLING MILL CO., MIDDLETOWN, OHIO

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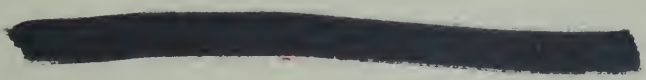

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Preface

WE ARE ISSUING THIS BULLETIN on account of the numerous requests we receive for copies of the methods used in our laboratories, especially those referring to the analysis of pure American Ingot Iron. Some of the methods are essentially as described in the standard text books, others are entirely new. We have not attempted to include all the elements existing in special steels. We refer the chemist to standard text books for the methods of analysis not herein described. This bulletin is intended as an aid to experienced chemists who are thoroughly conversant with the standard methods for the analysis of iron and steel. For the sake of brevity we have omitted details which are fully described in the text books, but in some cases where we considered it advisable we have given minute details.

We invite criticism and suggestions in reference to new or modifications of old methods, which will be duly credited to the author if published in our future bulletins.

Research Laboratory
The American Rolling Mill Company
Middletown, Ohio.

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METHODS OF ANALYSIS

Determination of Silicon

Dissolve 4.69 grams of the sample in a platinum dish, using 60 c. c. of nitric acid, 1.18 specific gravity, and 10 c. c. of sulphuric acid, 1.84 specific gravity. Evaporate to dense white fumes and allow to cool. Dissolve ferric sulphate in about 35 c. c. of hydrochloric acid, 1.20 specific gravity, dilute and filter. Filter on ashless paper and wash alternately with distilled water and dilute hydrochloric acid, 1.05 specific gravity, until free from iron. Ignite in platinum crucible, using a Meker burner with natural draft. Weigh residue and add about 1 c. c. of hydrofluoric acid and about 3 drops of concentrated sulphuric acid. Heat crucible carefully until acid has evaporated, then to full temperature of burner until iron has changed to oxide. Cool and weigh. The loss is silica. Each milligram equals .01% of silicon.

AMERICAN INGOT IRON

Clear Ferrite, Medium Grain, Absence of Slag and Gases

Determination of Sulphur

Dissolve 5 grams of the sample in 100 c. c. of hydrochloric acid, 1.10 specific gravity, contained in a 500 c. c. flask fitted with rubber stopper containing thistle tube and educt tube, passing through a reflux condenser to prevent acid distilling over. The educt tube dips almost to the bottom of a 10" x 1" test tube containing 50 c.c. of cadmium chloride solution. A low flame is applied and flask heated until all metal has dissolved and all gas has been driven out of flask, as is evidenced by the steam condensing in cadmium chloride tube. The contents of test tube are washed into an 800 c. c. beaker and sufficient water is added to bring the volume to 500 c. c. Add 5 c. c. of starch solution and 50 c. c. of hydrochloric acid, 1.20 specific gravity. The solution is then titrated with standard iodine solution to blue color.

The standard iodine solution is prepared by dissolving 8.4 grams of iodine and 20 grams of potassium iodide in 50 c. c. of distilled water. When iodine is in solution dilute to 2 liters and standardize with steel of known sulphur content. 1 c. c. should equal .01% of sulphur when using 5 grams.

The starch solution should be prepared fresh daily, using 1 gram of arrowroot mixed in

10 c. c. of cold distilled water, which is poured into 100 c. c. of boiling water and immediately removed from fire.

The cadmium chloride solution is prepared by dissolving 10 grams of cadmium chloride in 1500 c. c. of distilled water and 500 c. c. of .90 specific gravity ammonia.

Determination of Phosphorus

Dissolve 2 grams of the sample in 40 c. c. of nitric acid, 1.18 specific gravity, using a 300 c. c. Erlenmeyer flask. Heat on hot plate until metal is in solution and add 5 c. c. of saturated solution permanganate of potash. Boil until brown precipitate is formed. Now add a few c. c. of hydrochloric acid, 1.20 specific gravity, in sufficient amount to clear the solution by boiling a few minutes. Remove from hot plate, cool somewhat, and cautiously add ammonia, .90 specific gravity, shaking flask occasionally, until a heavy precipitate of ferric hydroxide is formed. Then add nitric acid, 1.42 specific gravity, shaking occasionally, until precipitate dissolves and a clear amber-colored solution is obtained. Heat or cool solution to 85° C. and add 50 c. c. of ammonium molybdate solution. Shake well and allow to stand at least ½ hour or until precipitate settles. Filter and wash with 2% nitric acid solution until free from iron, and finally with distilled water containing about 1 gram of potassium nitrate to liter until free from acid. Transfer filter and contents to tumbler containing 50 c. c. of boiled distilled water. Disintegrate paper with two stirring rods and add sufficient standard sodium hydroxide to dissolve the yellow precipitate and render the solution pink

when phenolphtalein indicator is added. Now run in standard nitric acid until pink color disappears.

The acid and alkali are about .15 normal. 1 c. c. is equal to about .01% of phosphorus when 2 grams are taken for analysis. The exact standardization of the acid and alkali solutions is made by the use of steel standards prepared by the U. S. Bureau of Standards.

1.18 specific gravity nitric acid can be prepared by adding 1 part of nitric acid, 1.42 specific gravity, to 2 parts of water.

Determination of Carbon

The determination of carbon in iron and steel is made by direct combustion, using a $\frac{3}{4}$"-bore x 30" silica tube contained in an electric furnace. We have found that a gas furnace is not reliable where the gas pressure fluctuates considerably. This is due to the danger of overheating and causes devitrification of the silica tube, with a consequent loss of carbon.

When determining traces of carbon the greatest care is necessary in order to secure accurate determinations.

The carbon dioxide is absorbed in potassium hydrate, and care must be taken to always weigh the bulbs under the same conditions. If the bulb contains air during the first weighing and oxygen during the second, the error in weighing will amount to several hundredths of a per cent.

We use a 6" x $\frac{1}{2}$" x $\frac{1}{2}$" platinum boat for holding the borings, and we can conveniently use 5 grams of the sample for a direct determination of carbon. The boat is covered for a depth of $\frac{1}{8}$" with 60-mesh alundum, on which the borings are placed; we then cover the borings completely with alundum and burn for 30 minutes at 950° C.

We have found that a short alundum thimble inserted in the $\frac{3}{4}$"-bore silica tube next to the stopper will prevent radiated heat from acting on the rubber stopper.

Carbon can also be determined by color as follows:

In determining carbon by color it is essential that the standard contain approximately the same per cent carbon as the sample, and also that the materials have had the same heat treatment. For the analysis of pure iron we furnish free a vial of standardized American Ingot Iron.

Dissolve $\frac{1}{2}$ gram of the sample and $\frac{1}{2}$ gram of the standard in 10 c. c. of nitric acid, 1.18 specific gravity, contained in separate 10" x 1" test tubes. Heat over Bunsen flame until metal has dissolved and tubes are free from brown fumes. Cool carefully and pour into carbon comparison tubes. Match color of standard with the color of sample by diluting with distilled water. Knowing the carbon content of standard the carbon of sample can be easily calculated.

Determination of Manganese

Titration Method

This is the bismuthate method as perfected by Professor D. J. Demorest, of the Ohio State University, which is substantially as follows:

Dissolve 1 gram of the sample in 45 c. c. of water and 15 c. c. of nitric acid, 1.42 specific gravity, and boil until nitrous fumes are gone. After cooling somewhat, sodium bismuthate is added, a little at a time, until the resulting permanganic acid or manganese dioxide persists after a few minutes boiling. Now 3 c. c. of a 5% solution of potassium nitrite is added to reduce the manganese compounds, and the solution is boiled a few minutes to expel the nitrous fumes. It is then cooled to tap water temperature. When cold, sodium bismuthate is added, a little at a time, while the solution is shaken, until about $\frac{1}{2}$ gram has been added. After settling a few minutes the solution is filtered by suction through asbestos on glass wool contained in a 3″ funnel (or an alundum crucible can be used). The filter is well washed with a 3% solution of nitric acid prepared from colorless acid. The permanganic acid is then titrated with standard sodium arsenite solution until the pink tinge just disappears. There

should not be a brownish color at the end. If there is, it indicates insufficient acid.

The sodium arsenite is prepared by dissolving 2 grams of arsenious acid in a hot solution of sodium carbonate, using sufficient sodium carbonate to completely dissolve the arsenious acid. It is then filtered through paper and diluted to 2 liters. 1 c. c. is approximately equal to .00025 gram of manganese. The solution is standardized against steel of known manganese content.

By Color

Dilute the solutions from carbon determination to 30 or 40 c. c. Remove 10 c. c. from each with a pipette, place in 10" x 1" test tubes and add to each 3 c. c. of nitric acid, 1.18 specific gravity. Bring to boil and add ½ gram of lead peroxide (free from manganese) to each and boil vigorously for 3 minutes. Cool and pour into 15 c. c. centrifuge tubes and separate the undissolved lead peroxide by centrifuging a few minutes. Decant clear solution into comparison tubes and dilute until colors match. If no centrifuge is available the lead peroxide can be filtered out on an alundum crucible or on an ignited asbestos filter either of the Gooch or cone type.

Determination of Chromium

This method is based on the fact that chromium and manganese are oxidized by sodium bismuthate in either nitric acid, sulphuric acid, or a mixture of nitric and sulphuric acids. Nitric acid alone is generally used, and only in rare instances will it be necessary to add sulphuric acid to facilitate the solution of the metal. Some manganese is oxidized to permanganic acid, which is decomposed by boiling, forming nitrate of manganese and manganese dioxide. The manganese dioxide is removed by filtration through asbestos, washing the asbestos well with a 3% solution of nitric acid. If any chromium is present it will be indicated by a yellow color in the filtrate.

Dissolve 3 grams of the sample in a mixture of 70 c. c. of water and 30 c. c. of nitric acid, 1.42 specific gravity. Boil until metal is in solution. Cool slightly and add 2 grams of sodium bismuthate, taking care to wash all bismuthate from the neck of the flask. Boil for 15 minutes, or until permanganic acid is decomposed. Filter with suction on asbestos supported by a tuft of glass wool in a 3″ glass funnel. Wash with 3% nitric acid. Cool to tap water temperature and dilute to 500 c. c. with distilled water. Add a measured excess of ferrous

ammonium sulphate solution until free from yellow tints. Titrate the excess with standard permanganate to faint pink color that persists for 30 seconds.

Titrate with permanganate 50 c. c. of ferrous ammonium sulphate containing the same amount of acid and water as the test.

The amount of ferrous ammonium sulphate oxidized by the chromic acid and measured in terms of permanganate is multiplied by the iron factor x .31, or 1 c. c. of tenth normal potassium permanganate equals .00173 gram of chromium.

The ferrous ammonium sulphate is prepared by dissolving 50 grams of the salt in 2 liters of 10% by volume of sulphuric acid. When this strength solution is used, 1 c. c. will equal about ½ c. c. of tenth normal permanganate.

The following table indicates the accuracy of the method, showing in some instances a very small per cent of chromium in the presence of a very high per cent of manganese:

Chromium Added	Chromium Found	Manganese
1.44	1.42	.67
1.44	1.48	.01
.37	.37	.38
.33	.35	.55
.29	.31	.75
.24	.26	.95
.12	.11	1.30
.04	.04	1.90

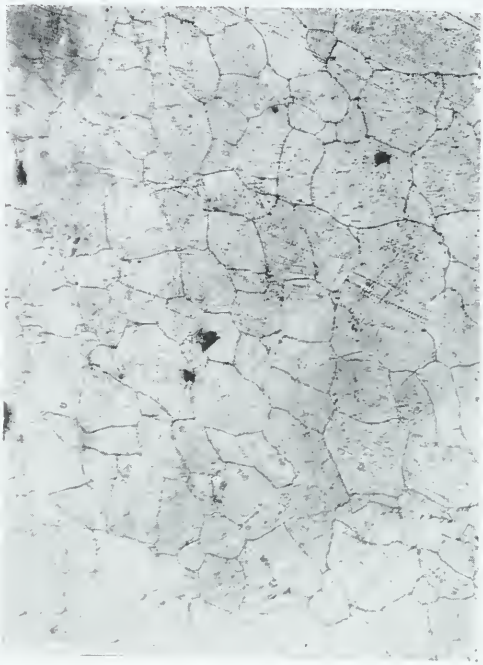
Determination of Vanadium

Vanadium can be determined in the same solution as the chromium as described by C. M. Johnson in his book "Chemical Analysis of Steel," which is essentially as follows:

To the flask containing the solution from the chromium determination, add 3 c. c. of a 1% solution of potassium ferricyanide. Standard ferrous ammonium sulphate is now added from a burette until 1 drop produces a green coloration.

A blank determination is made on steel free from vanadium. This blank varies from .4 to 1 c. c. of ferrous ammonium sulphate and should be deducted before making the calculations.

The amount of ferrous ammonium sulphate used in terms of potassium permanganate is equivalent to .00512 grams of vanadium for a tenth normal permanganate solution.



NORWAY IRON FROM NEWBURYPORT BRIDGE LINK

After 100 Years' Service. Clear Ferrite, Medium Grain
Very Little Slag and Free from Gases

Gravimetric Determination of Iron

Dissolve about 1 gram[1] of the sample, accurately weighed, in a 600 c. c. Jena glass beaker[2], on the water bath, using 25 c. c. of a 10% solution of hydrochloric acid. When solution is complete, add 200 c. c. of distilled water, heat on water bath to about 80° C. and pass a moderate current of hydrogen sulphide gas through the solution for 20 minutes. Remove from water bath, add 200 c. c. more of cold distilled water, and continue the stream of hydrogen sulphide for another 20 minutes, or until the solution is cold. Filter from the precipitate and wash thoroughly with hydrogen sulphide water containing a small amount of hydrochloric acid[3], collecting the filtrate in an 800 c. c. Jena glass beaker[2]. Test the residue

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1. By employing a power-driven centrifuge of large capacity the washing of precipitate can be perfectly and quickly performed largely by decantation, thereby enabling the operator to use a sample as large as 5 grams.

2. Beakers, funnels, watch glasses and glass rods must be thoroughly cleaned with warm concentrated hydrochloric acid before use, in order to prevent any foreign iron from entering the solutions.

3. The presence of acid is necessary to secure a perfect removal of iron from the residue and the filter paper. The hydrogen sulphide removes the following elements: silver, lead, mercury, gold, platinum, tin, antimony, arsenic, copper, cadmium, bismuth, molybdenum, tellurium, selenium, germanium, iridium, osmium, palladium, rhodium, ruthenium, tungsten, vanadium.

for iron[4]. Evaporate the filtrate on the water bath until the volume is reduced to about 200 c. c. and all the hydrogen sulphide is driven off. Then add 5 c. c. of concentrated nitric acid and 10 c. c. of concentrated hydrochloric acid[5], and heat to about 90° C. on the water bath and add a slight excess of warm dilute ammonia[6]. Allow the precipitate to settle, decant through a 15 ctm. ashless filter, transfer the precipitate to the filter[7] and wash with boiling water until 5 c. c. of the washings give no opalescence with silver nitrate[8]. (Collect the filtrate and the first washings in a clean 800 c. c. beaker and reserve for determination of manganese). Dry the ferric hydroxide precipitate at 95-100° C.[9] and then separate as perfectly as possible from the filter paper, in a room free from draught, placing the dry ferric hydroxide in a small porcelain dish on a white glazed paper. Cover the porcelain dish with a

4. If great accuracy is desired, the analysis should be rejected whenever more than traces of iron are detected in the sulphide residue.

5. The nitric acid is added to oxidize the iron, and the hydrochloric acid to secure sufficient ammonium chloride to keep zinc, cobalt, nickel and part of the manganese in solution. In nickel steels the precipitation should be repeated several times.

6. A large excess of ammonia is objectionable, as it causes some of the iron to become colloidal.

7. Ferric hydroxide adheres to the beaker and the glass rod. In order to remove this quantitatively, a few drops of concentrated nitric acid

watch glass and then ignite the filter paper in a weighed porcelain crucible. Transfer the precipitate from the porcelain dish to the crucible; cover with a platinum cover and ignite for 10 minutes over a Bunsen burner; remove the cover, incline the crucible slightly and ignite for another 10 minutes. Place in desiccator, cool and weigh. Repeat the ignition until the weight remains constant, taking care not to heat more than a few minutes at a time and not at too high temperature[10].

In the meantime evaporate the filtrate for the determination of manganese, to about 200 c. c. Add ammonia, heat to boiling and precipitate the manganese with a saturated solution of bromine. Boil for a few minutes, filter on a small ashless filter, ignite and weigh as $Mn_3 O_4$.

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are introduced into the beaker and by means of the glass rod all ferric hydroxide is easily brought in solution. After dilution with water the iron is reprecipitated with ammonia and transferred to the filter. Nitric acid is used in order to prevent introduction of chlorides (see 8).

8. When iron is precipitated with ammonia, small amounts of basic iron salts are always thrown down with the hydroxide. The amount and the composition of the basic salts vary according to the conditions. Thus in a solution of sulphate of iron larger amounts of basic salts are formed than from solutions of ferric nitrate or chloride. In a cold solution more basic salts are formed than when the solution is nearly boiling, and in addition the ferric hydroxide tends to assume a colloidal state, especially in the presence of a large excess of ammonia. Basic chloride of iron is volatile on ignition, hence the necessity of eliminating chlorides. Warm water decomposes chloride of iron, leaving the hydroxide free from chlorine.

This weight subtracted from the weight of Mn_3O_4 in the sample, calculated from the total manganese, gives the amount of Mn_3O_4 in the ferric oxide.

To determine silica in the ignited precipitate, transfer this to a small platinum dish and digest with concentrated hydrochloric acid on the water bath and evaporate to dryness[11]. Redissolve, dilute with hot water and filter from the silica on a small ashless filter. Wash with hot dilute hydrochloric acid and hot water until the silica is free from iron. Ignite in a platinum crucible, cool and weigh, and then evaporate with 2 drops of sulphuric and 1 c. c. of hydrofluoric acid; ignite, cool and weigh.

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9. The filter paper will become brittle if heated at a temperature above 100° C. Dry at least 10 hours. Heat gradually in crucible.

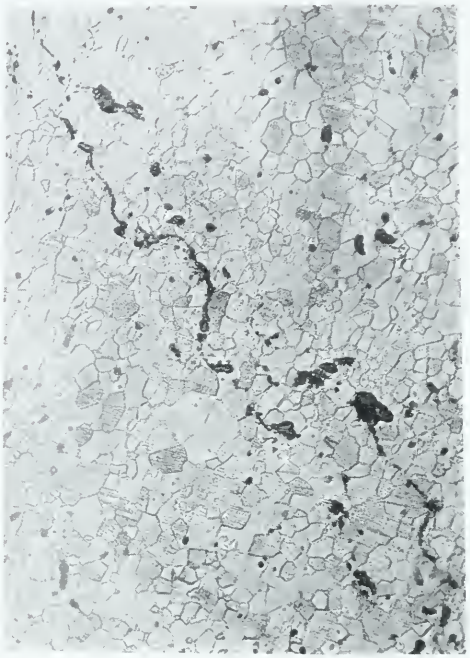
10. When ferric oxide is ignited at too high a temperature, some magnetic oxide of iron (Fe_3O_4) is always formed, causing low results. The formation of magnetic oxide takes place much more readily when the ignition is performed in a platinum crucible A convenient arrangement consists of placing a small porcelain crucible within a covered platinum crucible, whereby the contact with platinum is avoided and the danger of overheating greatly reduced ; at the same time the disadvantage of using a porcelain crucible alone is overcome.

11. If the oxide has been heated to a high temperature it is difficult to dissolve in concentrated hydrochloric acid. To secure solution in a reasonable length of time in cases when the oxide has been overheated, it is advisable to grind the oxide carefully in an agate mortar and then ignite it for a minute, reweigh and determine the silica on this portion and from the result calculate the silica in the original amount of oxide.

The difference between the two weighings then represents the amount of silica in the ferric oxide[1 2].

The total amount of silica, manganese oxide, chromic oxide, phosphoric acid and alumina (calculated from analysis), subtracted from the weight of impure ferric oxide, gives the weight of pure Fe_2O_3, which contains 69.94% iron (Fe=55.84).

12. In all exact gravimetric determinations of iron, allowance must be made for silica in the ferric oxide. Most steel and iron contain silicon, and considerable amounts are always dissolved from the glassware during the chemical operations. Glass is readily attacked by warm ammonia. Part of the silica is undoubtedly derived from the ammonia which ordinarily has been in contact with common glass. Most of the phosphorus is evolved during the solution in hydrochloric acid. If, however, the sample contains more than a few thousandths of 1% of phosphorus, the ferric oxide should be analyzed for phosphorus. This can be done in the filtrate from the determination of silica, and the amount found, figured to phosphoric anhydride, added to the other impurities.



STEEL
Showing Presence of Gases

Determination of Copper

Take the filtrate from the determination of sulphur and add ammonia, .90 specific gravity, until solution becomes light brown. If, however, any precipitate separates, add sufficient dilute hydrochloric acid to dissolve it, then add water to bring volume to about 400 c. c. Pass hydrogen sulphide gas into solution until it smells strongly of this gas. Filter and wash sulphides with hydrogen sulphide water. Open paper against side of funnel; add 20 c. c. of hot nitric acid, 1.18 specific gravity*, to paper in funnel, allowing same to run into original flask; wash paper with dilute hydrochloric acid, evaporate solution to about 15 c. c., remove from heat and add ammonia, .95 specific gravity, until iron is precipitated and solution smells of ammonia. Filter into a 100 c. c. Nessler tube and wash with hot water. The copper will be found in filtrate as a blue solution. To another Nessler tube add about 50 c. c. of distilled water and 5 c. c. of .90 specific gravity ammonia. Then add, from a burette, standard copper sulphate solution until the colors match when diluted to same volume.

*If stronger nitric acid is used it acts upon the filter paper, preventing all the iron from being precipitated with ammonia and producing an off-color difficult to match.

In material containing low copper there may be some difficulty in matching the colors, but this can be overcome by adding a few drops of a 10% solution of potassium ferro-cyanide and sufficient dilute sulphuric acid to faint acidity. The reddish brown colors are then compared.

The standard copper sulphate solution is prepared by dissolving 7.856 grams of crystallized copper sulphate in about 200 c. c. of distilled water and 10 c. c. of nitric acid, 1.42 specific gravity. The solution is then diluted to 1 liter. Each c. c. equals .04% copper when 5 grams have been taken for analysis.

Determination of Nitrogen

We use the Allen method perfected by Professor J. W. Langley. This method is based on the reaction by which the combined nitrogen in iron or steel is estimated as ammonia by the solution of the metal in hydrochloric acid.

The reagents required are:

Hydrochloric acid of 1.1 specific gravity, free from ammonia, which may be prepared by distilling pure hydrochloric acid gas into distilled water free from ammonia. To do this, take a large flask fitted with a rubber stopper carrying a separatory funnel tube and an evolution tube. Place in the flask strong hydrochloric acid, connect the evolution tube with a wash bottle connected with a bottle containing the distilled water. Admit strong sulphuric acid free from nitrous acid to the flask through the funnel tube, apply heat as required, and distil the gas into the prepared water.

Test the acid by admitting some of it into the distilling apparatus, described further on, and distilling it from an excess of pure caustic soda, or determine the amount of ammonia in a portion of hydrochloric acid of 1.1 specific gravity, and use the amount found as a correction.

Solution of caustic soda, made by dissolving 300 grams of fused caustic soda in 500 c. c. of water, and digesting it for 24 hours at 50° C. on a copper-zinc couple prepared by rolling together about 6 square inches each of zinc and copper foil.

Nessler reagent. Dissolve 35 grams of potassium iodide in a small quantity of distilled water, and add a strong solution of mercuric chloride little by little, shaking after each addition, until the red precipitate formed dissolves. Finally the precipitate formed will fail to dissolve; then stop the addition of the mercury salt and filter. Add to the filtrate 120 grams of caustic soda dissolved in a small amount of water, and dilute until the entire solution measures 1 liter. Add to this 5 c. c. of a saturated aqueous solution of mercuric chloride, mix thoroughly, allow the precipitate formed to settle, and decant or siphon off the clear liquid into a glass-stoppered bottle.

Standard ammonia solution. Dissolve .0382 gram of ammonium chloride in 1 liter of water. 1 c. c. of this solution will equal .01 milligram of nitrogen.

Distilled water free from ammonia. If the ordinary distilled water contains ammonia, redistil it, reject the first portions coming over, and use the subsequent portions, which will be

found free from ammonia. Several glass cylinders of colorless glass of about 160 c. c. capacity are required.

The best form of distilling apparatus consists of an Erlenmeyer flask of about 1500 c. c. capacity, with a rubber stopper carrying a separatory funnel tube and an evolution tube, the latter connected with a condensing tube, around which passes a constant stream of cold water. The inside tube where it issues from the condenser should be sufficiently high to dip into one of the glass cylinders placed on the working table.

The determination of nitrogen is made as follows: Place 40 c. c. of the caustic soda, which has been treated with the copper-zinc couple, in the Erlenmeyer flask, add 500 c. c. of water and about 25 grams of tinfoil to prevent bumping, and distil until the distillate gives no reaction with the Nessler reagent. While this part of the operation is in progress dissolve 3 grams of the carefully washed drillings in 30 c. c. of the prepared hydrochloric acid, using heat if necessary. Transfer the solution to the bulb of the separatory funnel tube, and when the soda solution is free from ammonia, very slowly drop the ferrous chloride solution into the boiling solution in the flask. When about 50 c. c. of water has been collected in

the cylinder, remove it and substitute another cylinder. Place 1½ c. c. of the Nessler reagent in a cylinder, dilute the distillate to 100 c. c. with the special distilled water and pour it into the cylinder containing the Nessler reagent. Take another cylinder, place therein 1½ c. c. of the Nessler reagent and 100 c. c. of the special distilled water to which 1 c. c. of the ammonium chloride solution has been added, and compare the colors of the solutions in the two cylinders. If the solution in the cylinder containing the ammonium chloride solution is lighter in color than that in the cylinder containing the distillate, place 1½ c. c. of the Nessler reagent in another cylinder, pour into it 100 c. c. of water containing 2 or more c. c. of the ammonium chloride solution, and repeat this operation until the colors of the solutions in the two cylinders correspond after standing about 10 minutes. When about 100 c. c. have distilled into the second cylinder, replace it and test as before. Continue the distillation until the water comes over free from ammonia, then add together the number of c. c. of ammonia solution used, divide the sum by 3, and each .01 milligram will be equal to .001% of nitrogen in the steel.

Determination of Hydrogen

The method for determining hydrogen by heating the metal in a partial vacuum and measuring the liberated hydrogen is very laborious. The hydrogen existing as ammonia would not be estimated by the above method.

Hydrogen is kinetically the most active element. Some of the hydrogen is liberated by drilling, so that it is necessary to work with the metal in a single piece, if possible. However, if the metal is in strips, several can be used for an analysis.

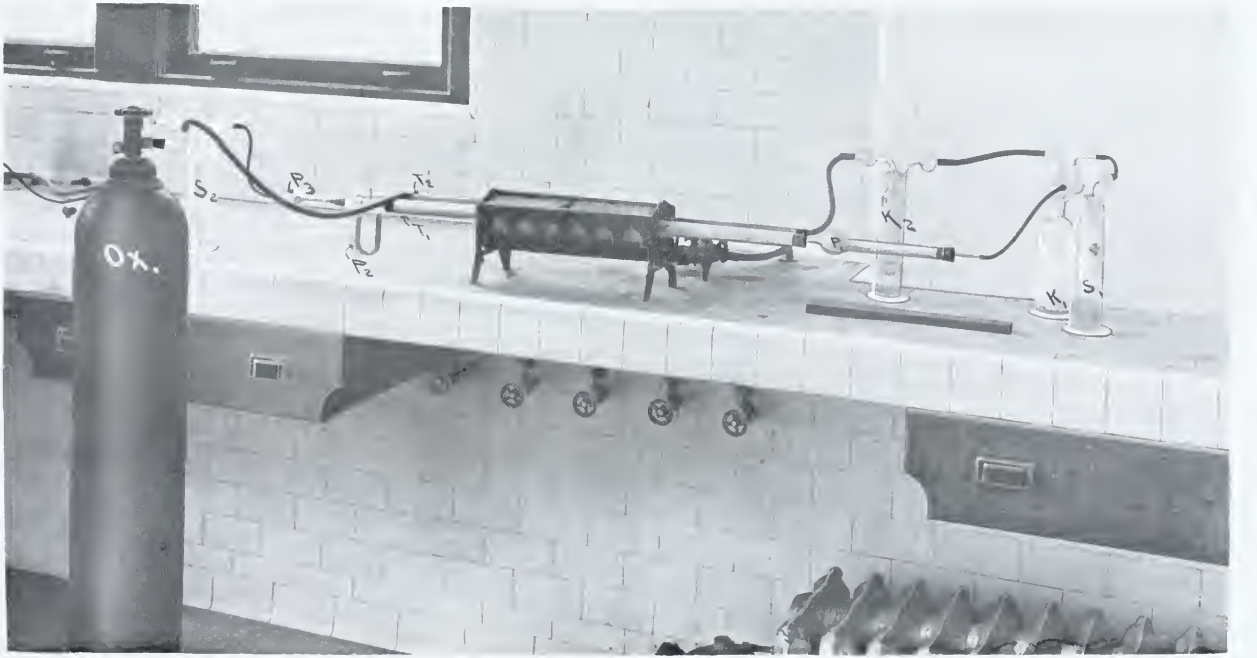
The method is based on the fact that hydrogen is liberated by heating the metal to a red heat in an atmosphere of oxygen. The hydrogen is oxidized to water, which is absorbed in phosphoric anhydride.

The apparatus used consists of a 12" gas blast furnace in which is placed a 30" x $\frac{3}{4}$" silica tube, and on top of this tube is placed a 30" x $\frac{1}{4}$" silica tube. The silica tubes each have a 6" roll of platinum gauze to act as a catalyzer. The oxygen gas passes through the $\frac{1}{4}$" silica tube (T-2) at the rate of 100 c. c. per minute, and the impurities are thus oxidized. The gas then passes through a wash bottle containing a strong solution of caustic potash (K-2), then through a bottle containing stick

caustic potash (K), then through a bottle containing concentrated sulphuric acid (S-1), and finally through a tube containing phosphoric anhydride opened up with glass wool (P-1). The purified oxygen then enters the $^{3}/_{4}$" silica tube (T-1), where it combines with the liberated hydrogen, forming water, which is swept into the 4" glass stop-cock U-tube containing phosphoric anhydride opened up with glass wool (P-2). This tube is weighed and connected with a tube of phosphoric acid opened up with glass wool (P-3) used as a trap. The gas finally passes through a solution of concentrated sulphuric acid, which is used to show that the gas is passing through the apparatus.

The silica tubes should be at a red heat and oxygen passing through the apparatus at the rate of 100 c. c. per minute. Place in either a clay boat, or one of platinum containing ignited alundum, the sample in as large pieces as are available, using from 10 to 40 grams for a determination.

Weigh U-tube (P-2) and connect direct with stopper to $^{3}/_{4}$" silica tube. Remove stopper and tube (P-1) and insert the boat into the red-hot zone of the silica tube. Connect tube (P-1) with silica tube and continue passing the oxygen gas through the apparatus at the rate


Ox.
S2
P3
P2
K2
K1
S1

of 100 c. c. per minute for 30 minutes. At the end of this time disconnect weighing tube (P-2) and connect with a suitable aspirator, so as to suck out the oxygen and replace it with dry air. A suitable aspirator consists of a 1-gallon aspirator bottle filled with water. The upper tubular is guarded with a calcic chloride tube to which the weighing tube is connected, and the other side of weighing tube is connected with a phosphoric acid tube followed by a washing bottle containing concentrated sulphuric acid.

The aspirator may be roughly calibrated by allowing 500 c. c. of water to run out of the lower tubular of the aspirator. This is a sufficient amount of dry air to thoroughly displace all the oxygen.

The glass stop-cocks of weighing tube are now closed and weighing tube disconnected from guard tube and placed in a desiccator for 15 minutes before being weighed. One-ninth of the increased weight of the tube is hydrogen.

It is absolutely necessary to run a blank determination using the same amount of oxygen as for a regular test, and it must also be run for the same length of time. The blank should never exceed 1 milligram. This blank is derived from the oxidation of the rubber con-

nections, hence the necessity for using a definite amount of oxygen for a definite length of time.

In charging the weighing tube with phosphoric anhydride, place about a gram on glass wool, fold the glass wool over the phosphoric acid and insert into the tube. Repeat until the tube is full. Use great care to remove all specks of phosphoric acid from the induct and educt tubes of weighing tube.

The temperature of the silica tubes must be regulated so that the sample does not absorb all the oxygen. If the temperature is too high this will occur and no oxygen will pass through the apparatus.

It is not necessary to burn all the metal to oxide; the time that would be required to do so would be prohibitive on large samples.

The following results have been obtained by these methods:

Material	Sul.	Phos.	Carb.	Mang.	Cop.	Sil.	Oxy.	Nit.	Hyd.
16 ga. American Ingot Iron	.020	.001	.010	.025	.072	.002	.021	.0032	.0003
16 ga. Steel	.072	.113	.120	.350	.020	.009	.047	.0071	.0012
16 ga. American Ingot Iron	.017	.006	.015	.020	.028	.002	.023	.0030	.0007
16 ga. American Ingot Iron	.015	.003	.010	.010	.028	.005	.035	.0031	.0009
Electrolytic Iron, very brittle	.003	.003	.005	.000	.000	.000	.025	.0030	.0310
Electrolytic Iron, after heating in hydrogen, very malleable	.003	.003	.005	.000	.000	.000	.000	.0030	.0006

Determination of Oxygen in Iron and Steel*

The determination of oxygen in iron and steel has not received sufficient attention in the United States. The text books on iron and steel analysis which are in common use in this country do not include methods of analysis for oxygen content of irons and steels. This is probably due to the fact that in the ordinary steel-making processes ferro-manganese can be freely used, so that it is assumed that the percentage of oxygen in the steel rarely reaches or exceeds the danger point. As a matter of fact, steel often carries more oxygen than it should, as can be seen by referring to Table II.

In the manufacture of iron of very high purity in basic open-hearth furnaces the oxygen content has to be very carefully watched, or the product may be overburned and contain an excess of oxygen.

A number of methods for the determination of oxygen have been proposed, among which may be mentioned: (1) Heating the sample in a stream of dry chlorine; (2) Dissolving the sample in special solvents such as copper sulphate or bromine; (3) Combustion of the

*Paper published by Allerton S. Cushman, Journal of Industrial and Engineering Chemistry, June, 1911.

sample in the form of borings in pure dry hydrogen.

The latter method, which is due to Ledebur,* is the only one that has proved reliable. In Ledebur's original method, the sample is given a preliminary combustion in pure nitrogen in order to burn off the last traces of impurities and to get rid of all hydrocarbons. If the preliminary heating in nitrogen is dispensed with, the results will be slightly higher, but it is probable that for general work sufficiently accurate results can be obtained if the sample is carefully prepared for the combustion in hydrogen.

Samples

The samples should consist of fine borings or shavings from a milling machine. The drill or machine tool should be scrupulously clean and free from all traces of oil or dirt, and should be geared to run slowly so as not to heat the sample while it is being cut. Lack of careful attention to this point will lead to high results owing to surface oxidation of the fine particles of the drillings.

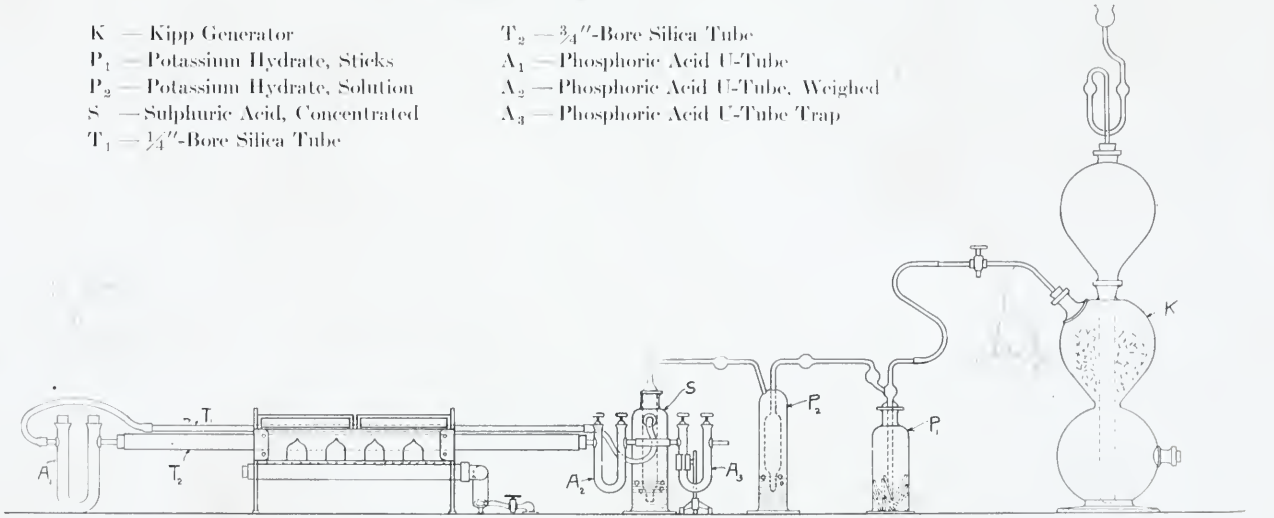
Apparatus

The apparatus used in making the oxygen determination is shown in illustration B-740.

*Leitfaden fur Eisenhuttenlaboratorien. Vieweg und Sohn, Braunschweig, 6 Auflage, 1903, S. 122.

DENOTATIONS

K — Kipp Generator
P_1 — Potassium Hydrate, Sticks
P_2 — Potassium Hydrate, Solution
S — Sulphuric Acid, Concentrated
T_1 — ¼″-Bore Silica Tube
T_2 — ¾″-Bore Silica Tube
A_1 — Phosphoric Acid U-Tube
A_2 — Phosphoric Acid U-Tube, Weighed
A_3 — Phosphoric Acid U-Tube Trap



B-710

A 1-gallon Kipp generator is used for generating the hydrogen. It should be charged with drillings of pure iron or mossy zinc, and dilute hydrochloric acid (1-1). Steel turnings should not be used in the generator, as the object is to generate the purest possible hydrogen. Hydrochloric is preferable to sulphuric acid. After its formation the hydrogen is purified and dried by passing through the usual train as shown in the figure. It passes first over stick potash, and next through a 30% potash solution. This solution in the second bottle should be renewed as soon as it shows a tinge of yellow due to the presence of sulphides. The hydrogen next passes through concentrated sulphuric acid to dry it, and then enters a silica tube with $\frac{1}{4}$" bore, 30" in length, which contains a roll of platinum gauze. The $\frac{1}{4}$" tube lies on top of a 1" x 30" fused silica tube contained in a suitable 12" gas blast furnace.

The object of the preliminary heating over platinum foil is to free the hydrogen from the small quantity of oxygen which it always contains. If this precaution is not taken, the results will be too high. The water formed in the small-bore silica tube is caught in a U-tube shown in the figure, which contains phosphoric anhydride opened up with glass wool.

This drying tube has rubber stoppers. The connection is made with pure gum tubing and is permanent, the sample being introduced from the opposite end of the combustion tube.

Blanks should be run from time to time to make sure that the apparatus is in good order and everything working properly. Samples should not be introduced into or removed from the combustion tube when it is more than hand hot, but silica tubes may be quickly cooled with perfect safety by turning off the gas and allowing the cold air blast to play on the tube.

Method

Weigh 20 to 30 grams of finely divided borings into a nickel or platinum boat $\frac{1}{2}$" x $\frac{1}{2}$" x 6". The boat with its charge is quickly inserted into the combustion tube and pushed to the middle zone by means of a rod of suitable length. The stream of hydrogen should be passing freely when the tube is opened for the insertion of the sample. After the stopper is replaced, the weighing tube and guard tube are finally connected up with pure gum tubing. The weighing tube is a 4" U-tube, with ground glass stoppers, containing phosphoric anhydride opened up with glass wool. The guard or trap tube is similarly

charged and is intended to prevent the drawing back of moisture from the air of the laboratory. After the apparatus is all connected up and in good order, the pure dry hydrogen should be allowed to sweep through a few minutes until all air is removed from the entire system. The gas is then lighted, the blast turned on and the temperature quickly run up to a bright red heat, about 850° C. This heat is maintained for 30 minutes, while the hydrogen is passing through the apparatus at the brisk rate of about 100 c. c. per minute. After the combustion is completed the gas is turned off the furnace, leaving the blast playing upon the hot tube. The stream of hydrogen should continue to pass until the tube is cool enough to bear the hand upon it.

Immediately after the tube is cool enough, the weighing tube, with its guard tube, is disconnected and connected with a suitable aspirator, so as to suck out the hydrogen gas and replace it with dry air. A suitable aspirator consists of a 1-gallon aspirator bottle filled with water. The upper tubular of the bottle is guarded with a calcic chloride tube to which the weighing tube is connected. A gas washing bottle containing concentrated sulphuric acid follows the phosphoric acid tube which is connected to the other side of the weighing tube.

The aspirator may be roughly calibrated by allowing about 500 c. c. of water to run out of the lower tubular of the aspirator. A sufficient quantity of perfectly dry air is drawn through to thoroughly displace all the hydrogen. After all is ready, the weighed tube is closed by its glass stop-cocks, disconnected from its guard tube and placed in a desiccator for 15 minutes before being weighed. Eight-ninths of the increased weight of the tube is oxygen. The blanks on the apparatus establish the average correction to be subtracted from the weight found. The correction on an apparatus in good order should not exceed 3 milligrams. On damp days the blank is usually a little higher than when the air is dry.

In charging the weighing tube with phosphoric anhydride and glass wool, take care to remove any specks of phosphoric acid from the upper portions of the tube.

The following points should be given careful attention in order to attain the highest degree of accuracy:

Samples must be clean, absolutely dry and free from oil. They should be cut, preferably with a milling machine tool running at a low rate of speed. The samples must not heat in cutting. Sheet samples are first cleaned from oxide on an emery wheel, avoiding heating as

much as possible. The sheet should be milled on the edge.

Whenever possible, samples should be cut from bars which are first cleaned by a superficial cut with the milling tool. Extreme care must be taken in the preparation of the sample.

The entire apparatus must be kept to the top notch of cleanliness, tightness and general good order. Blanks should be run frequently. Analysis should be in duplicate whenever the results are to be used as a basis for specification. The most extreme care should be taken to exclude all oxygen from the sample and apparatus except that which it is the object of the method to determine. When determining oxygen in pure iron, the silver white iron residues from the boat should be reserved for charging the Kipp hydrogen generator.

The claim has been made that the modified Ledebur method as described above does not determine total oxygen, as the oxides of manganese, aluminum, silicon, titanium and other possible constituents or impurities of steel are not reduced by combustion in hydrogen. This statement is true, but it is our belief that only the oxygen combined with iron measures the state of deoxidation of the metal, and that a method of analysis which determines total oxygen in steel without differentiation is quite valueless. In addition to this it must be remembered that American Ingot Iron is essentially free from oxides unreducible by combustion in hydrogen, so that there could be little or no difference between total oxygen and oxygen by the Ledebur method in the case of a metal of a high degree of purity.

In the following table are given a number of oxygen determinations:

Table I

Material	Sil.	Sul.	Phos.	Carb.	Mang.	Nit.	Oxy.
American Ingot Iron (01107) Sheet[1]	.003	.021	.002	.01	Trace	.0050	.027
American Ingot Iron (02007) Sheet[1]	.003	.018	.004	.015	.020	.0060	.014
Electrolytic Sheet Iron, by Electrolysis	000	.001	.005	.006	.000	.0030	.032
American Ingot Iron Chain Link	Trace	.022	.008	.015	.025	.0063	.032
Norway Iron Chain Link, 100 years old[2]	.019	.007	.032	.050	.000	.0047	.058
Puddled Iron 1½″ Bar, broke in service	.056	.047	.140	.065	.070	.....	.855
Norway Iron Piano Wire	.007	.006	.012	.025	.020	.....	050
Puddled Iron Chain Link	.077	.009	.072	.010	.010	.....	.145
Puddled Iron Sheet[3]	.077	.020	.074	.015	.010	.....	.283

1. See photomicrograph, page 10.
2. See photomicrograph, page 22.
3. See photomicrograph, page 56.

In this table a number of oxygen determinations in steels are given. These results were obtained by Romanoff*, working with the Ledebur method.

Table II

No.	Carbon	Phosphorus	Manganese	Sulphur	Oxygen	Specific Gravity
496	.107	.04	.57	.03	.19	7.79
478	.25	.01	.54	.03	.15	7.85
572	.25	.02	.55	.04	.26	7.72
516	.10	.04	.50	.07	.11	7.92
503	.10	.08	.55	.06	.25	7.84
526	.095	.02	.37	.06	.22	7.78
540	.10	.04	.48	.07	.16	7.82
547	.095	.04	.49	.05	.11	....
550	.105	.05	.49	.03	.14	....
551	.09	.06	.32	.04	.16	7.87
553	.095	.08	.39	.04	.13	7.80
555	.10	.05	.44	.03	.13	7.87
570	.095	.02	...	...	.03	8.08
081	.105	.06	.46	...	.09	7.97
583	.10	.04	.42	.03	.02	8.08

*Stahl und Eisen, March 15, 1899.

Analysis of Tin and Terne Plate and Lead Coated Sheets

Several samples exactly 2" x 2" should be taken for analysis. Clean thoroughly with carbon tetrachloride or gasoline and weigh. A 400 c. c. Jena glass beaker has been found the most convenient for this test. We have found that 20 c. c. of concentrated sulphuric acid is sufficient for each 2" x 2" piece. If 4 pieces are taken, however, 60 c. c. of sulphuric acid will be sufficient. Place the requisite amount of acid in the beaker and heat to at least 250° C. Wrap a stiff platinum or nickel wire around one of the 2" x 2" pieces so that it can be placed in the acid in a horizontal position. Immerse the piece in the hot acid for exactly 1 minute. Transfer the piece to another 400 c. c. beaker containing 25 c. c. of distilled water and rub the surface of the sample while washing with about 50 c. c. more distilled water, using a wash bottle for this purpose. Dry the sample thoroughly and reweigh. The loss in weight represents the coating and some iron. Repeat this operation for each sample, collecting all rinsings in a beaker, and reserve for analysis.

The iron which has dissolved is determined as follows: The sulphuric acid is carefully

poured into the beaker containing the washings from the 2" x 2" pieces. This solution is cooled and poured into a volumetric flask. 25% by volume of concentrated hydrochloric acid is added and the flask filled to the mark with distilled water. If 4 pieces have been taken for analysis, a 500 c. c. volumetric flask will be required. Use a proportionately smaller flask if less than 4 samples are analyzed. Mix thoroughly, and transfer 100 c. c. of the solution to a 300 c. c. Erlenmeyer flask. Add a solution of tenth normal permanganate until iron and tin are oxidized, which is indicated by the appearance of a permanent straw color. No account is taken of the amount of permanganate used. Heat to boiling and reduce carefully with stannous chloride. Cool and pour into a 1000 c. c. beaker containing 500 c. c. of distilled water and 25 c. c. of saturated solution of mercuric chloride, stir vigorously, add 50 c. c. of the titrating mixture of phosphoric acid and manganese sulphate, and titrate with tenth normal permanganate to pink color. The amount of iron which has thus been determined is subtracted from the total weight lost in sulphuric acid; the remainder is coating. It is very often unnecessary to make an analysis of the coating, the object being merely to determine the weight.

There are several ways of expressing the weight of the coating; we prefer to express

it in ounces per square foot. By knowing the number of square feet in a box of tin plate or a case of terne plate there is no confusion in converting the ounces per square foot to pounds per box or case. The coating on tin plate is sometimes expressed in pounds per box of 112 sheets 14" x 20". This figure can be obtained by multiplying the number of grams of coating of each 2" x 2" piece by 17.29. If it is desired to express the coating on terne plate in pounds per case of 112 sheets 20" x 28", then multiply the number of grams of coating on each 2" x 2" piece by 34.57. The average of the several pieces represents the weight of coating.

If the determination of tin and lead is desired in the sheet, proceed as follows: Place another 100 c. c. of the sulphuric acid solution containing the coating in a 300 c. c. Erlenmeyer flask. Should or should not any lead sulphate be removed with this 100 c. c., does not influence the accuracy of the tin analysis. Add 50 c. c. of concentrated hydrochloric acid and 1 gram of finely ground antimony. Connect flask with a 1-hole stopper containing a glass tube bent twice at right angles, the end of which projects into a beaker of water. Boil 5 minutes and replace the beaker containing the water with one containing an 8% solution of bicar-

bonate of soda prepared from boiled distilled water. Remove flask from hot plate and allow the soda water to flow back into the flask while cooling same with tap water. When cold, add a few c. c. of starch solution and titrate to permanent blue color with tenth normal iodine solution. 1 c. c. equals .00595 gram of tin. The amount of tin found is subtracted from the weight of coating which has been determined by loss in sulphuric acid, and after the iron correction has been made the remainder is lead. An example of a regular analysis of terne plate is as follows:

Piece 2" x 2" weighs.....7.563 grams
Same after stripped in acid weighs.6.721 grams

Loss coating plus iron weighs..... .842 grams

Sulphuric acid and washings were diluted to 200 c. c., 100 c. c. of which was titrated for iron. This required 10.1 c. c. of tenth normal permanganate, which is equivalent to .0564 gram of iron. As half of the solution only was taken for analysis, it is necessary to multiply by 2, which is equivalent to .1128 gram iron.

Total weight of coating plus iron... .8420 gram
Weight of iron dissolved..... .1128 gram

Coating..... .7292 gram

.7292 x 34.57 = 25.21 pounds coating per case of 112 sheets 20" x 28".

The tin was then determined in 50 c. c. of the sulphuric acid solution. This required 7.8 c. c. of tenth normal iodine.

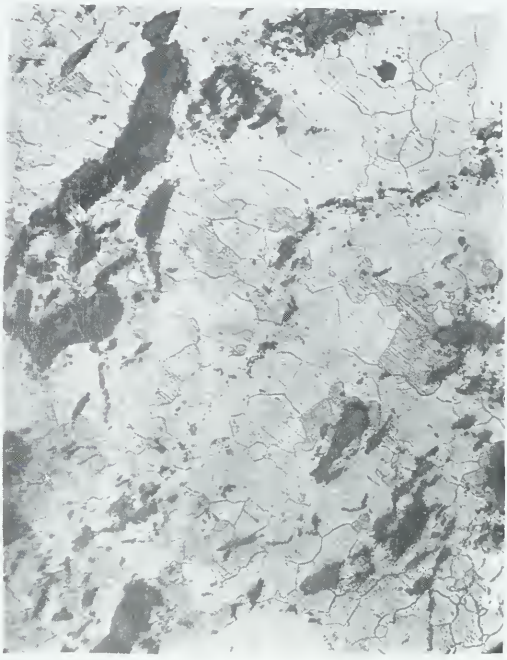
$$7.8 \times .00595 \times 4 = .1856 \text{ gram tin.}$$

	Coating	Tin	Lead
Grams	.7292 —	.1856 =	.5436

By knowing the original weight of coating and the weight of tin and lead in this coating, it is a very simple matter to determine the percentage of each element.

In the determination of tin in tin plate it is only necessary to determine the loss in sulphuric acid, and then to determine the iron which has been dissolved, the remainder being tin.

Heavily coated lead sheets may require twice as much acid and a temperature of 300° C. to completely remove the coating in 1 minute.



PUDDLED IRON
Showing Grains of Ferrite and Slag Inclusions

Test to Indicate Whether Metal is Iron or Steel

In making this test without the use of a balance the following table can be used when metal is in sheet form and the gauge is known. These weights represent the number of grams in 1 square inch:

Gauge	Grams Sq. Inch	Gauge	Grams Sq. Inch	Gauge	Grams Sq. Inch
12	14.00	17	7.10	22	4.00
13	12.00	18	6.40	23	3.61
14	10.00	19	5.66	24	3.20
15	9.00	20	4 82	25	2.80
16	8.00	21	4.41	26	2.41

As an illustration, suppose we have 16 gauge material. A square inch weighs 8 grams, hence a strip $\frac{1}{8}$" x 1" weighs approximately 1 gram, or $\frac{1}{4}$" x $\frac{1}{2}$" would also equal 1 gram. If the sheet is galvanized the coating need not be removed before making the test.

Take equal portions of American Ingot Iron and sample to be tested, equal to $\frac{1}{2}$ gram, and place in separate 10" x 1" test tubes. Add to each tube 15 c. c.[1] of dilute nitric acid, 1.18 specific gravity[2]. Place a test tube in holder, using care to incline the tube away from

spectators while being heated with an alcohol lamp. The metal will disappear in a few minutes, but continue heating until no more brown fumes are given off. Allow solution to cool and heat the other tube in same manner and cool. The solutions can be compared at this point, the darker one containing the highest per cent of carbon.

To each test add ½ gram sodium bismuthate[3] and agitate for a few minutes. Then add sufficient water to half fill the tubes and mix thoroughly. Allow the tubes to rest for several minutes until the undissolved sodium bismuthate settles. By comparing the clear solutions, American Ingot Iron will show a light pink color, while steel will yield a purple color due to manganese present.

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1. If no graduate is available, the volume of acid can be estimated by noting the depth of 1″ diameter tube; each inch is equal to about 10 c. c.

2. 1.18 specific gravity nitric acid can be prepared by adding 1 part 1.42 specific gravity nitric acid to 2 parts water.

3. ½ gram sodium bismuthate is about the maximum amount that can be placed on a dime.

Pin Hole Test—Lead Coated, Tin and Terne Plate

Dr. Allerton S. Cushman has devised a very simple test to determine the number of pin holes per square foot. The test consists of exposing a full sized sheet to the action of distilled water. The pin holes appear as rust spots.

The four sides of the sheet are bent so as to make a pan 1" deep. The pan is thoroughly cleaned with several applications of gasoline and then flooded to a depth of $\frac{1}{2}$" with distilled water. After 1 week's exposure the water is removed and the pin holes are counted.

Hydrochloric Acid Method for Determining Ounces of Spelter per Square Foot

Take about 10 samples of exactly 2″ x 2″ from each sheet, weigh all samples together and place, one at a time, in 200 c. c. of concentrated hydrochloric acid for exactly 1 minute, turning samples over during test. Remove samples from acid, wash carefully, scrub, dry and weigh. The loss in weight is coating, which, divided by the number of pieces used and multiplied by 1.27, gives ounces per square foot.

This method is especially recommended for the determination of spelter on American Ingot Iron on account of the slight solubility of pure iron in hydrochloric acid.

If the spelter is to be determined upon Bessemer or other steel readily acted upon by hydrochloric acid, this method is not recommended, and the lead acetate method should be used.

Lead Acetate Method for the Determination of Spelter Coating

The basis of this test is as follows:

When a zinc-coated iron article is placed in this solution at ordinary temperatures, the zinc passes into solution, and an equivalent amount of metallic lead is precipitated in a loosely adherent form upon the specimen. The reaction is retarded by the precipitation of the lead, and, therefore, when a heavily galvanized piece is being tested, this lead must be periodically removed. The acetate solution can be used to measure by time just as the Preece copper sulphate solution, if desired. Should lead plate on, it is not easily confounded with the bright iron when exposed. The uncovering of the iron can be readily detected.

The solution used for making this test is prepared by dissolving 400 grams of crystallized lead acetate in 1 liter of water. When dissolved, add 4 grams of finely powdered litharge and agitate until most of it has dissolved. The solution is allowed to settle and the clear portion decanted for use.

Ordinary glass tumblers have been found very satisfactory to use in making this test, as they are the right diameter to enable the

sample to be maintained in an upright position without supports.

The samples should be taken from various parts of the sheet. Use several 2" x 2" pieces cut accurately to $\frac{1}{64}$". Weigh the samples together and submerge separately, for 3 minutes, in tumblers containing solution of lead acetate. The samples are then taken out and the adherent lead removed with a stiff brush or steel spatula. A burnishing action should be avoided, as under some conditions closely adherent lead will be plated out on the iron. Repeat the 3-minute immersions in the lead acetate solutions until a bright surface is exposed. Four 3-minute immersions are usually sufficient. Wash specimens in water, dry and weigh. The loss in grams represents the coating, which, divided by the number of 2" x 2" pieces used and multiplied by 1.27, gives the number of ounces of coating per square foot.

The Republican Publishing Company
Hamilton, Ohio, U. S. A.